Supporting information

Nanopalladium-Catalyzed Conjugate Reduction of Michael Acceptors – Application in Flow

Anuja Nagendiran, a,b,c Henrik Sørensen, a,b,d Magnus J Johansson, c,d Cheuk-Wai Tai, e Jan-E. Bäckvall b,c

jeb@organ.su.se

a Authors contributed equally to this work

b Department of Organic Chemistry, Arrhenius Laboratory, Stockholm University, SE-106 91 Stockholm, Sweden

c Berzelii Centre EXSELENT on Porous Materials, Arrhenius Laboratory, Stockholm University, SE-106 91 Stockholm, Sweden

d AstraZeneca R&D, Innovative Medicines, Cardiovascular and Metabolic Disorders, Medicinal Chemistry, Pepparedsleden 1, SE-431 83 Mölndal, Sweden

e Department of Materials and Environmental Chemistry, Arrhenius Laboratory, Stockholm University, SE-106 91 Stockholm, Sweden

1. General information

2. Typical procedure for hydrogenation in flow

3. Detailed experimental description for substrates

4. Schematic view of the flow devices

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1. General Information

The substrates and the solvents were purchased from commercial sources and used as supplied. The silica based Pd\textsuperscript{0}-AmP-MCF was synthesized as previously reported\textsuperscript{1} and analysed with inductively coupled plasma (ICP-OES) for determination of palladium content, which was measured to 12wt%. ICP-OES was also used for determination of palladium content in the leaching tests. For H-Cube\textsuperscript{®} hydrogenations, the catalyst (Pd\textsuperscript{0}-AmP-MCF) was sent to Thalesnano\textsuperscript{2} for filling of cartridges. Each H-Cube\textsuperscript{®} cartridge (30 x 4 mm) was loaded with 70-80 mg of Pd\textsuperscript{0}-AmP-MCF. For the Vapourtec reactor (Omnifit 6 mm column) 226-242 mg of Pd\textsuperscript{0}-AmP-MCF was packed between plugs of celite at each end in a column. NMR spectra were recorded at 298 K using CDCl\textsubscript{3}. Chemical shifts are reported in ppm relative to solvent peaks (\textsuperscript{1}H: CDCl\textsubscript{3} δ 7.26 and \textsuperscript{13}C: CDCl\textsubscript{3} δ 77.0) with multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet, app. = apparent), coupling constants (in Hz) and integration. For \textsuperscript{19}F NMR the spectrum was referenced by using the deuterium signal of CDCl\textsubscript{3}.\textsuperscript{3} GC-MS analyses were performed on an Agilent 7890GC/5975CMSD spectrometer equipped with a HP-5MS, 30 m x 0.25 mm, 0.25 μm column. For detection of the molecular mass either electron impact or field ionization was used. 1,3,5-Trimethoxybenzene was added to the reaction mixture before hydrogenation for reactions quantified by analysis of \textsuperscript{1}H NMR spectra. For preparative experiments, benzyl benzoate has been used as reference for assay of the eluents after removal of the solvent/purification.

2. Typical procedures for hydrogenation in flow

**General procedure for hydrogenation using H-Cube®:**

The cartridge (filled with Pd\textsuperscript{0}-AmP-MCF) was equilibrated with EtOAc for 3 minutes under the same conditions as the hydrogenation of the substrate. A 0.1 M solution of the substrate in EtOAc was then hydrogenated at a flow rate of 1.5 mL/min, a temperature of 20 °C, and the hydrogen setting at full H\textsubscript{2} (30 mL H\textsubscript{2}/min) unless otherwise stated. For completion of the hydrogenation pure solvent was flushed through the cartridge for another 3 minutes under the same conditions. For assay experiments the eluent was analyzed by \textsuperscript{1}H NMR and for preparative experiments the solvent was removed *in vacuo* to give the products indicated in the respective experiments.
General procedure for hydrogenation using Vapourtec reactor:
The catalyst was equilibrated with EtOAc for 3 minutes under the same conditions as the hydrogenation of the substrate. A 1 M solution of the substrate in EtOAc was then passed through the column at the H₂-pressure, temperature and flow indicated for each substrate. Unless otherwise indicated a 40 psi back pressure regulator was fitted after the column. After consumption of the substrate solution the reactor was washed for 3 minutes with EtOAc under the same conditions as for hydrogenation of the substrate.

3. Detailed experimental description for substrates

3-Phenylpropanal (1b) (run in H-Cube®): A solution of cinnamaldehyde (5.33 g, 40.3 mmol) and 1,3,5-trimethoxybenzene (754 mg, 4.48 mmol) in EtOAc (403 mL) was prepared. After hydrogenation according to the general procedure the eluent was analyzed by \(^1H\) NMR. A ratio of 85% aldehyde 1b and 15% alcohol 1c was found.

3-Phenylpropanal (1b) (run in Vapourtec): A 1 M solution was prepared by dissolving cinnamaldehyde (3.53 g, 26.7 mmol) in EtOAc (27 mL). The solution was passed through a Vapourtec reactor packed with 236 mg Pd⁰-AmP-MCF (0.266 mmol Pd) at 1 mL/min, 50 °C, and pressure of 4 bar. Full consumption of the starting material and a ratio of 6% 1c and 94% 1b was obtained (by \(^1H\) NMR). Evaporation of the solvent gave 3.39 g oil which was purified over a 50 g column of silica gel using 0-40% EtOAc in heptane as eluent. After evaporation of the solvent 2.27 g (63%) 1b. \(^1H\) NMR (500 MHz, CDCl₃) δ 2.76 – 2.84 (m, 2H), 2.98 (app. t, \(J = 7.6\) Hz, 2H), 7.19 – 7.25 (m, 3H), 7.28 – 7.34 (m, 2H), 9.84 (t, \(J = 1.4\) Hz, 1H). \(^13C\) NMR (126 MHz, CDCl₃) δ 201.50, 140.30, 128.58, 128.26, 126.28, 45.26, 28.11. The analytical data were in agreement with those previously reported.⁴
3-Phenylpropanal (1b) (leaching test, run in Vapourtec): A solution of cinnamaldehyde (24.7 g, 187 mmol) in 162 ml EtOAc was passed through an (equilibrated) Vapourtec reactor freshly packed with 226 mg Pd$^0$-AmP-MCF (0.255 mmol Pd) at 1 mL/min, 50 °C, and a H$_2$ pressure of 4 bar. After collection of the eluent the solvent was removed to give 25.22 g residue consisting of a mixture of 1a (3%), 1b (90%), and 1c (7%). Analysis of this material showed a palladium content of 6 ppb.

![Chemical structure of 2a and 2b](image)

4-Phenylbutan-2-one (2b) (run in H-Cube®): A 0.1 M solution of 4-phenyl-buten-2-one (0.209 g, 1.43 mmol) was made in EtOAc (14.4 mL). After hydrogenation according to the general procedure the eluent was collected and the solvent was removed in vacuo to give 212 mg (98%) 2b as a colorless oil.

$^1$H NMR (500 MHz, CDCl$_3$) δ 2.14 (s, 3H), 2.76 (t, $J = 7.7$ Hz, 2H), 2.90 (t, $J = 7.7$ Hz, 2H), 7.16 – 7.23 (m, 3H), 7.26 – 7.31 (m, 2H). $^{13}$C NMR (126 MHz, CDCl$_3$) δ 207.88, 140.96, 128.46, 128.26, 126.08, 45.16, 30.05, 29.72. The analytical data were in agreement with those previously reported.$^3$

![Chemical structure of 3a and 3b](image)

1,3-Diphenylpropan-1-one (3b) (run in H-Cube®): A solution of (E)-chalcone (0.299 g, 1.43 mmol) in EtOAc (14 mL) was hydrogenated according to the general procedure. After removal of the solvent the product was obtained as an oil (301 mg, 100% combined yield) containing 10 mol% 1,3-diphenylpropan-1-ol.

$^1$H NMR (500 MHz, CDCl$_3$) δ 3.04 - 3.11 (app. t, $J = 7.7$ Hz, 2H); 3.31 (app. t, $J = 7.7$ Hz, 2H), 7.21 (m, 1H), 7.26 - 7.34 (m, 4H), 7.46 (app. t, $J = 7.7$ Hz, 2H), 7.53 - 7.59 (m, 1H),
7.94 - 7.99 (m, 2H). $^{13}$C NMR (126 MHz, CDCl$_3$) $\delta$ 199.36, 141.44, 137.00, 133.20, 128.75, 128.67, 128.57, 128.18, 126.28, 40.61, 30.29. The analytical data were in agreement with those previously reported.$^5$

![Diagram](image)

3-Phenylpropanoic acid (4b) (run in H-Cube®): A 0.1 M solution of cinnamic acid (0.206 g, 1.39 mmol) was made in EtOAc (14 mL). After hydrogenation according to the general procedure removal of the solvent gave 202 mg (97%) of the product as a colorless oil.

$^1$H NMR (500 MHz, CDCl$_3$) $\delta$ 2.69 (app. t, $J = 7.8$ Hz, 2H), 2.97 (app. t, $J = 7.8$ Hz, 2H), 7.19 - 7.24 (m, 3H), 7.28 - 7.33 (m, 2H). $^{13}$C NMR (126 MHz, CDCl$_3$) $\delta$ 178.58, 140.11, 128.54, 128.24, 126.36, 35.48, 30.56. The analytical data were in agreement with those previously reported.$^3$

![Diagram](image)

Methyl 3-phenylpropanoate (5b) (run in H-Cube®): A 0.1 M solution of methyl cinnamate (0.235 g, 1.45 mmol) was prepared in EtOAc (14 mL). After hydrogenation according to the general procedure the eluent was collected and the solvent was evaporated to give 236 mg (99%) 5b as an oil.

$^1$H NMR (500 MHz, CDCl$_3$) $\delta$ 2.64 (t, $J = 8.0$ Hz, 2H), 2.96 (t, $J = 8.0$ Hz, 2H), 3.67 (s, 3H), 7.16 - 7.23 (m, 3H), 7.27 - 7.33 (m, 2H). $^{13}$C NMR (126 MHz, CDCl$_3$) $\delta$ 173.31, 140.49, 128.48, 128.24, 126.24, 51.60, 35.69, 30.93. The analytical data were in agreement with those previously reported.$^6$

![Diagram](image)
**3-Phenylpropanenitrile (6b)** (run in H-Cube®): A 0.1 M solution of cinnaminitrile (0.187 g, 1.45 mmol) was made in EtOAc (14.5 mL). After hydrogenation at a flow of 1.4 mL/min and a temperature of 30 °C the solvent was removed *in vacuo* to give 189 mg (99%) oil. 

$^1$H NMR (500 MHz, CDCl$_3$) δ 2.63 (t, $J$ = 7.4 Hz, 2H), 2.97 (t, $J$ = 7.4 Hz, 2H), 7.22 – 7.3 (m, 3H), 7.31 – 7.37 (m, 2H). $^{13}$C NMR (126 MHz, CDCl$_3$) δ 138.18, 129.02, 128.39, 127.38, 119.25, 31.73, 19.51. The analytical data were in agreement with those previously reported.$^3$

![Chemical Structure](image)

**Hexanal (7b)** (run in H-Cube®): A 0.1 M solution of ($E$)-hex-2-enal (0.496g, 5.06 mmol) and 1,3,5-trimethoxybenzene (81.1 mg, 0.48 mmol) was made in EtOAc (51 mL). After hydrogenation at 30 °C the eluent was analyzed by $^1$H NMR. A yield of 94% was indicated by assay.

**Hexanal (7b)** (run in Vapourtec): A column containing 226 mg Pd$^0$-AmP-MCF (0.255 mmol Pd) and a 75 psi back pressure regulator fitted after the column was used. A 1 M solution of ($E$)-hex-2-enal (1.07 g, 10.9 mmol) in EtOAc (11 mL) was hydrogenated at ~6 bar in the Vapourtec reactor at a flow rate of 1 mL/min and a temperature of 50 °C. The eluent was analyzed by $^1$H and $^{13}$C NMR, indicating complete conversion to hexanal.

$^1$H NMR (500 MHz, CDCl$_3$) δ 0.88 (t, $J$ = 7.0 Hz, 3H), 1.30 (m, 4H), 1.54 – 1.68 (m, 2H), 2.39 (td, $J$ = 7.4, 1.9 Hz, 2H), 9.74 (t, $J$ = 1.9 Hz, 1H). $^{13}$C NMR (151 MHz, CDCl$_3$) δ 202.55, 43.75, 31.23, 22.32, 21.66, 13.71. The analytical data were in agreement with those previously reported.$^7$

![Chemical Structure](image)

**Methyl 4-oxopentanoate (8b)** (run in H-Cube®): A 0.1 M solution of ($E$)-methyl 4-oxopent-2-enoate (0.315 g, 2.46 mmol) in EtOAc (24 mL) was prepared. After hydrogenation according to the general procedure evaporation of the solvent gave 7b (310 mg, 97%) as an oil.
1H NMR (500 MHz, CDCl₃) δ 2.20 (s, 3H), 2.59 (t, J = 6.6 Hz, 2H), 2.76 (t, J = 6.6 Hz, 2H), 3.68 (s, 3H). 13C NMR (126 MHz, CDCl₃) δ 206.54, 173.13, 51.72, 37.88, 29.78, 27.67.

The analytical data were in agreement with those previously reported.⁸

![Diagram](image.png)

**Butyronitrile (9b)** (run in H-Cube®): A 0.1 M solution of (E)- and (Z)-but-2-enenitrile (0.150 g, 2.23 mmol) (ratio about 2:1) and 1,3,5-trimethoxybenzene (52.5 mg, 0.31 mmol) in EtOAc (22 mL) was prepared. After hydrogenation according to the general procedure the eluent was analyzed by ¹H NMR, indicating full conversion of starting material to 9b.

1H NMR (500 MHz, CDCl₃) δ 1.05 (t, J = 7.4 Hz, 3H), 1.67 (q, J = 7.2 Hz, 2H), 2.29 (t, J = 7.1 Hz, 2H). 13C NMR (126 MHz, CDCl₃) δ 119.12, 18.80, 18.52, 12.80.

The identity of this material was confirmed by comparison with authentic material (¹³C NMR and ¹H NMR).

![Diagram](image.png)

**4,4,4-Trifluorobutanenitrile (10b)** (run in H-Cube®): A solution of (E)-4,4,4-trifluorobut-2-enenitrile (0.242 g, 2.00 mmol) and 1,3,5-trimethoxybenzene (68.7 mg, 0.408 mmol) was prepared in EtOAc (20 ml) and hydrogenated according to the general procedure and analyzed, 100% conversion to 10b was indicated by assay.

Preparative (run in H-Cube®): A 0.1 M solution of (E)-4,4,4-trifluorobut-2-enenitrile (0.3054 g, 2.52 mmol) was made in EtOAc (25 mL). After hydrogenation according to the general procedure the eluent was collected and the solvent was evaporated to give 201 mg colorless oil with an assay of 77%.

1H NMR (500 MHz, CDCl₃) δ 2.46-2.56 (m, 2H), 2.64 (t, J = 7.7 Hz, 2H). ¹⁹F NMR (471 MHz, CDCl₃) δ -67.40 (s, 3F). ¹³C NMR (126 MHz, CDCl₃) δ 125.26, 116.88, (q, J = 276.7 Hz), 30.38 11.20 (q, J = 31.2 Hz). The identity of this material was confirmed by comparison with commercial material (¹H NMR, ¹⁹F NMR, and ¹³C NMR).
3-(Dimethylamino)propanenitrile (11b) (run in H-Cube®): A 0.1 M solution of N,N-dimethylaminoacetonitrile (0.190 g, 1.97 mmol) and 1,3,5-trimethoxybenzene (88.4 mg, 0.53 mmol) was made in EtOAc (20 mL). $^1$H NMR analysis of the eluent after hydrogenation according to the general procedure showed that 4% of the material had been consumed. A very weak signal corresponding to the expected mass could be observed in the GC-MS trace but $^1$H NMR indicated starting material only.

3-(Dimethylamino)propan-1-ol (12c) (run in H-Cube®): A solution of (E)-3-(dimethylamino)acrylaldehyde (0.566 g, 5.70 mmol) and 1,3,5-trimethoxybenzene (82.5 mg, 0.49 mmol) was made in EtOAc (57 mL). Hydrogenation at 60 °C indicated 70% 3-(dimethylamino)propan-1-ol (by assay) and 30% starting material.

Preparative (run in H-Cube®): A 0.1 M solution of (E)-3-(dimethylamino)acrylaldehyde (0.244 g, assay 91%) was prepared in EtOAc (22 mL). Hydrogenation at 60 °C. The solvent was removed in vacuo to give 153 mg colorless oil. The assay showed a purity of 77%, indicating an effective yield of 51%.

$^1$H NMR (600 MHz, CDCl$_3$) $\delta$ 1.57 – 1.74 (m, 2H), 2.23 (s, 6H), 2.51 (t, $J = 5.9$ Hz, 2H), 3.76 (t, $J = 5.5$ Hz, 2H). $^{13}$C NMR (151 MHz, CDCl$_3$) $\delta$ 64.70, 60.15, 45.55, 28.04.

The identity of this material was confirmed by comparison with commercial material ($^{13}$C-NMR and $^1$H-NMR).
2-Isopropyl-5-methylcyclohexanone (menthone/isomenthone, 13b) (run in H-Cube®): A 0.1 M solution of 1.59 g (10.5 mmol) 6-isopropyl-3-methylcyclohex-2-enone in EtOAc (106 mL) was prepared and hydrogenated at 50 °C. The solvent was removed in vacuo to give 1.53 g (95%) the product as a colorless oil. $^1$H NMR analysis showed two isomeric ketones (95%, the mixture consisting mainly of isomenthone) and 2-isopropyl-5-methylphenol (5%). The analytical data were in agreement with those previously reported.  

2-Isopropyl-5-methylcyclohexanone (menthone/isomenthone, 13b) (run in Vapourtec): A solution of 6-isopropyl-3-methylcyclohex-2-enone (4.76 g, 31.3 mmol) in EtOAc (31 mL) was prepared and passed at 30 °C through a column of Pd$^0$-AmP-MCF (242 mg, 0.273 mmol) with a hydrogen pressure of 4.3 bar and a flow rate of 1 mL/min. Removal of the solvent gave 4.56 g colorless oil which consisted of a 80:15 (by GC) mixture of cis- and trans-isomeric ketones (isomenthone/menthone), 2-isopropyl-5-methylphenol (3%), and starting material (2%).

The same setup but run at a temperature of 50 °C gave a mixture of the cis- and trans-isomeric ketones (78%) and 2-isopropyl-5-methylphenol (22%).

$^1$H NMR (500 MHz, CDCl$_3$) $\delta$ 0.85 (m), 0.94 (m), 1.00 (m), 1.48 (m), 1.64 – 1.79 (m), 1.9 – 2.22 (m), 2.29 (m). $^{13}$C NMR (126 MHz, CDCl$_3$) $\delta$ 214.58, 212.40, 57.19, 55.89, 50.87, 48.03, 35.46, 34.39, 33.93, 29.43, 27.86, 26.91, 26.85, 25.89, 22.28, 21.45, 21.21, 20.89, 19.88, 18.69.

Cyclohexanone (14b) (run in H-Cube®): A solution of cyclohex-2-enone (0.238 g, 2.47 mmol) and 1,3,5-trimethoxybenzene (43.6 mg, 0.26 mmol) was made in EtOAc (25 mL). Hydrogenation according to the general procedure indicated 100% yield by assay.
**Cyclohexanone (14b)** (run in Vapourtec): A solution of cyclohex-2-enone (0.97 g, 10 mmol) in EtOAc (10 mL) was prepared and passed at 20 °C through a column of 236 mg Pd⁰-AmP-MCF (0.266 mmol Pd) with a hydrogen pressure of ~3 bar and a flow of 0.5 mL/min. ¹H NMR Analysis of the eluent showed 99% cyclohexanone and 0.5% phenol. The same setup using a flow of 1 mL/min resulted in 98% cyclohexanone and 2% phenol in the eluent. The same setup using a flow of 1 mL/min and a temperature of 50 °C resulted in 60% cyclohexanone and 40% phenol in the eluent.

**Cyclohexanone (14b)** (leaching test, run in H-Cube®): A solution of cyclohex-2-enone (237 mg, 2.47 mmol) in EtOAc (26 mL) was subjected to the hydrogenation conditions indicated in the general procedure. Analysis of the eluent showed a palladium content of < 2 ppb. ¹H NMR (500 MHz, CDCl₃) δ 1.70 (m, 2H), 1.83 (m, 4H), 2.30 (t, J = 6.7 Hz, 4H). ¹³C NMR (126 MHz, CDCl₃) δ 211.06, 41.56, 26.69, 24.68. The analytical data were in agreement with those previously reported.³

**Stability test**

**3-Phenylpropanal (1b)** (Stability test of Pd⁰-AmP-MCF in cartridge, run in H-Cube® with limited H₂): On day 1 a 0.1 M stock solution of cinnamaldehyde (3.96 g, 30 mmol) and 1,3,5-trimethoxybenzene (5.05 g, 30 mmol) was made in EtOAc (300 ml), on day 2 a 0.1 M stock solution of cinnamaldehyde (13.3 g, 101 mmol) and 1,3,5-trimethoxybenzene (1.70 g, 10.1 mmol) was made in EtOAc (1000 ml). At the start of the hydrogenations the system was primed with the resulting substrate solution at a flow of 1 mL/min, the pressure regulator set at 10 bar (controlled H₂ mode), and the temperature set at 20 °C. The eluent was sampled directly from the H-Cube® for ¹H NMR analysis after the run time indicated. The yield of product is given in relation to the internal standard. No other products could be observed in the eluents by ¹H NMR.
3-Phenylpropanal (1b) (Stability test of Pd\(^0\)-AmP-MCF in cartridge, run in H-Cube® at 50 bar): A 0.1 M stock solution of cinnamaldehyde (13.2 g, 99.6 mmol) and 1,3,5-trimethoxybenzene (1.22 g, 7.26 mmol) was made in EtOAc (1 liter). The system was primed with the resulting substrate solution at a flow of 3 mL/min for 5 minutes with no hydrogen pressure. Subsequently, the pressure regulator was set at 50 bar, the H\(_2\) setting in controlled mode, the temperature was set at 20 °C, and the flow was set at 1 mL/min. At all times full consumption of starting material was observed. The eluent was sampled directly from the H-Cube® for \(^1\)H NMR analysis after the run time indicated. The yield of product is given in relation to the internal standard. No other products could be observed in the eluents by \(^1\)H NMR.
Figure 2. Stability test for Pd⁰-AmP-MCF over time, when the hydrogenation was run at 50 bar pressure of H₂.

Figure 3. HAADF-STEM (a) and TEM Images (b and c) of unused Pd⁰-AmP-MCF on different scale.

Figure 4. HAADF-STEM (a) and TEM Images (b and c) of used Pd⁰-AmP-MCF on different scale.
Schematic view of the flow devices

**H-Cube®**

![Schematic view of H-Cube®](image)

*Figure 5. H-Cube® with Pre-packed cartridge with 70 mg Pd⁰-AmP-MCF (12 wt% Pd).*

**Vapourtec**

![Schematic view of Vapourtec](image)

*Figure 6. Schematic view of Vapourtec with 226 mg Pd⁰-AmP-MCF (12 wt% Pd).*

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2 ThalesNano Nanotechnology Inc., H-1031 Budapest,Zahony utca 7, Hungary


8 Izquierdo, J.; Rodríguez, S., González, F. V. *Org. Lett.,* **2011**, *13*(15), 3856-3859

Frequency: 500.13MHz (nmr13)
Spinner: 74
Experiment Name: Proton_day
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#Scans: 4000
Solvent: CDCl3
Parameters:
Date: Wed Apr 02 13:20:32 CEST 2014

ethyl acetate

ethyl acetate

ethyl acetate

ethyl acetate

10b
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Solvent: CDCl3
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Frequency: 150.90MHz (nmr7)
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Solvent: CDCl3
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#Scans: 16
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Parameters:
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Extension:

13b
cis/trans
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Spinner: 57
Experiment Name: Carbon_night
#Scans: 4000
Solvent: CDCl3
Parameters:
Date: Wed Mar 26 16:27:27 CET 2014
Extension:

cis/trans

13b
Frequency: 500.13MHz (nmr13)
Spinner: 44
Experiment Name: Proton_day
#Scans: 16
Solvent: CDCl3
Parameters:
Date: Mon Mar 24 17:24:37 CET 2014
Extension: 500ul_min

EtOAc

EtOAc

14b
Frequency: 125.76MHz (nmr13)
Spinner: 32
Experiment Name: Carbon_night
#Scans: 4000
Solvent: CDCl3
Parameters:
Date: Thu Apr 24 16:16:05 CEST 2014
Extension: 500ul